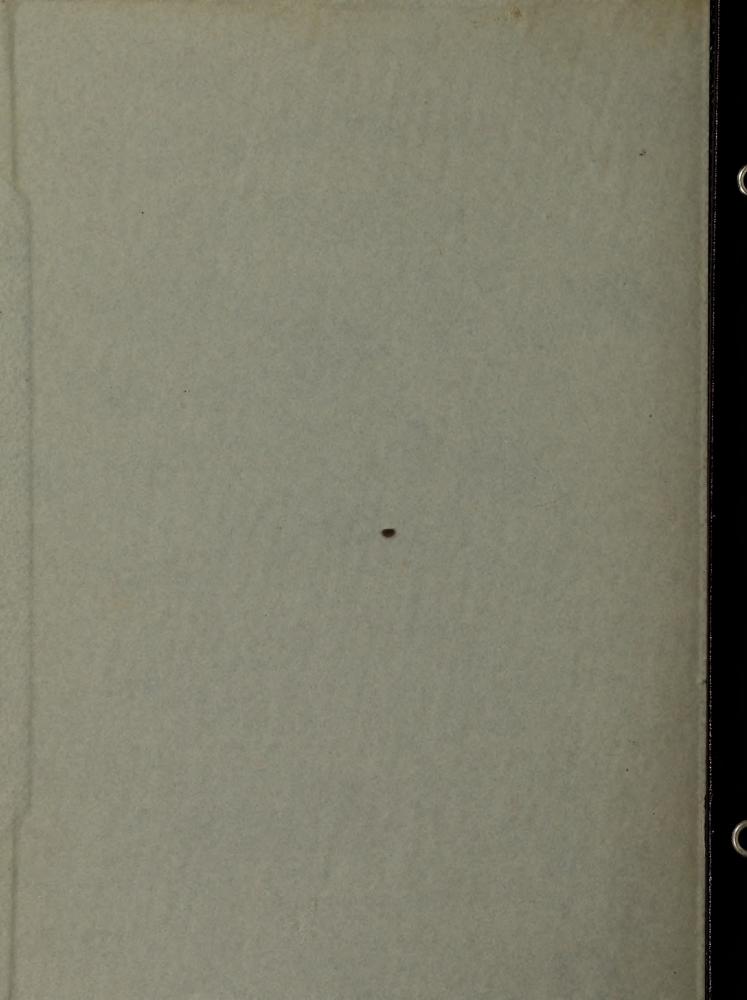
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BOSTON UNIVERSITY GRADUATE SCHOOL

Thesis

THE LEAD-URANIUM RATIO OF A CRYSTAL OF WILBERFORCE, ONTARIO,
URANINITE

by

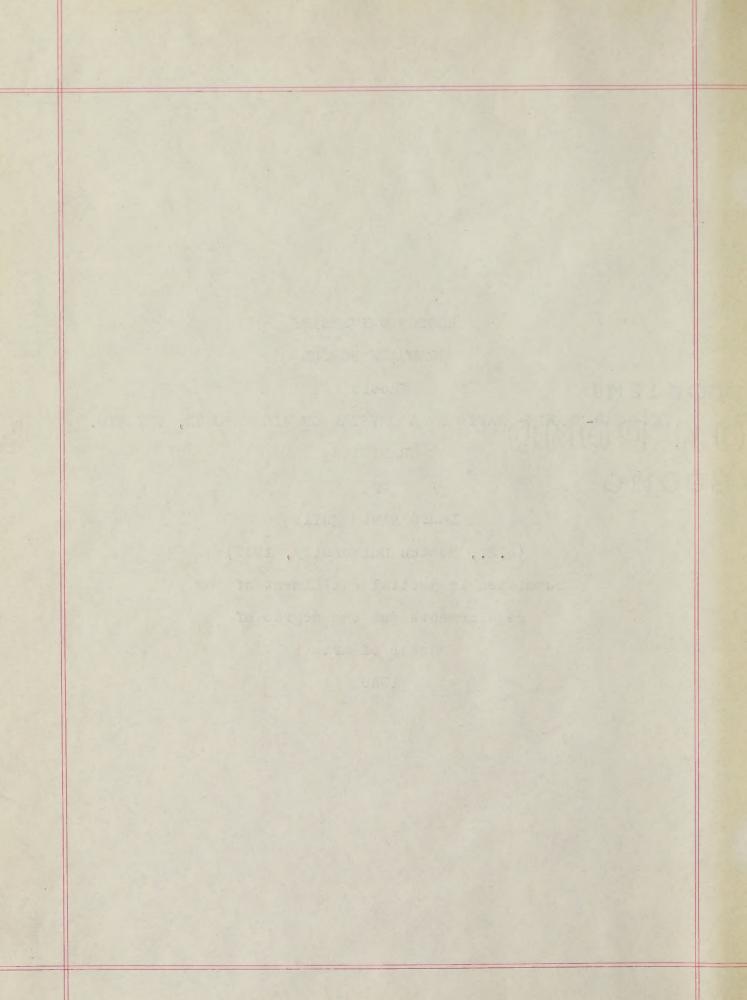
Laura Alvin Yuill

(B.S., Boston University, 1935)

submitted in partial fulfilment of the requirements for the degree of

Master of Arts

1936



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INDEXED OUTLINE

			Page
I.	Introduction		
II.	Possible Methods of Determining the Age of the Earth		1
	a,	Geological Method Based on Thickness of Strata	1
	ъ.	Age of the Ocean	- 2
	c.	Method Based on Astronomical Data	3
	d.	Method Based on Radioactive Disintegration	3
III.		ad Method of Determining the Age of active Minerals	7
	a.	Radioactivity and the Parentage of Radiogenic Lead	8
	b.	Derivation of Age Formula	11
	C.	Requirements of the Lead Method	14
	d.	Possible sources of error in the Lead Method	18
IV.	The Wi	lberforce Uraninite	21
	a.	Description of Deposits	21
	b.	Results Obtained by Other Workers	23
V.	Experi	mental Procedure	30
	a.	Standardization of Weights	31
	ъ.	Method of Weighing	36
	С.	Purification of Reagents	38
	d.	Division of Crystal	41

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		,I
		.II
	a. Geological Method Rance on Thickness of Strate	
8.	b. Age of the dosan	
	odelinod saged on Astronomical Data	
	d. Mathod Raged on Redicactive Disinte-	
		, III
	a. Madiosotivity and the Parentage of Madiogenic Lead	
	b, Verivavion of Age Formula	
	c. Requirements of the Lead Method	
	d. Possible so more of error in the	
		.VI
	a. Description of Deposits	
	b. Resolve Obtained by GV er Wurkere	
		.7
	a. Standardization of Weights	
	natified to boiled to	
	o. Farification of Heagonto	
	d. Division of Crystell	

<u>P</u>	age
e. Analysis for Lead, Uranium, and Thorium	44
f. Separation of Thorium and Uranium	51
g. Determination of Uranium	57
h. Determination of Thorium	61
VI. Experimental Results	62
/II. Summary	65

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INTRODUCTION

Since the turn of the present century and the discovery of radioactivity, there has been a great deal of work done in determining the age of the earth by means of data obtained from radioactive disintegration. This work has been done by chemical methods for the most part. However, these methods have not proven to be correct in every way, and it is the purpose of this piece of work to probe further into the present methods and see whether or not they are entirely trustworthy.

The possibility of preferential leaching has been recognized as a possible source of error in these determinations for some time. The present research should indicate whether or not such leaching does occur, and if it does, how errors due to this cause can best be avoided.

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POSSIBLE METHODS OF DETERMINING THE AGE OF THE EARTH

At the beginning of the twentieth century, there were three distinct estimates of the age of the earth. All were of the same order of magnitude, but all had been determined by different methods. These three estimates were G. H. Darwin's of 57 million years; Lord Kelvin's of 20-40 million years; and Joly's of 80-90 million years. With the discovery of radio-activity the very foundations upon which these estimates rested were destroyed, but at the same time a new way of determining the age of the earth presented itself. However, as a result of these new age determinations, it was found that the age was from 10-20 times as great as had previously been supposed. 1

The oldest scientific method of determining the extent of geologic time is based on a method which takes into account and measures the thickness of the strata that have accumulated during that time. However, even after the thickness of the strata has been determined, there is the problem of how to

1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
Research Council" as Bulletin 80. p.1

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1. "The Age of the Marth" Enapp, Schuchert, Estatik, Molmis, and Troms. Published by "The Dati onal Research Council" as Bulletin 30. p.1

convert this thickness into time. A mean rate of deposition really cannot be determined and the best that can possibly be hoped for is a mean rate for each basin. The most recent work on determining the age of the earth by the thickness of the strata, however, seems to indicate that it may really be possible to get some fairly accurate results by this method. \(^1\)

Another method of determining the extent of geologic time is based on the amount of sodium in the ocean. If the total amount of sodium in the ocean is divided by the amount washed in each year, a figure, "the age of the ocean", would be obtained. However the whole method rests on two rather large assumptions. First, it is assumed that the rate at which sodium has been washed into the ocean has been constant throughout geologic time. Second, it is assumed that the sodium has steadily accumulated in the ocean. Actually it is known that both assumptions are untrue. This leaves us with the conclusion that this method gives an estimate which would,

1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
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at best, be of the right order of magnitude. 1

A third method of determining the extent of geologic time is based on astronomical data. However, there are no known methods using astronomical data alone for estimating the age of the earth. About all that can be done in this method is arrive at an approximation of the right order of magnitude.²

The fourth method of determining the extent of geologic time is based on data compiled from radio-active disintegration. Under this heading there are at least three different ways of determining the age of radioactive minerals. First there is the method which makes use of pleochroic halos. Mineralogists have known for some time that in certain varieties of mica there are peculiar spherical regions of discoloration called pleochroic halos. In thin sections of the mica, these are seen as a series of distinct rings. The coloration is due to alpha particles emitted by a small grain of radioactive material at

- "The Age of the Earth" Knapp, Schuchert, Kevarik, Holmes, and Brown. Published by "The National Research Council" as Bulletin 80. pp. 65 ff.
- 2. Ibid pp. 40 ff.

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2. Ibid pp. 40 ff.

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Joly studied pleochroic halos in micas of varying ages and found that in the older ones, the radii of the halos was greater. This suggested a method of determining the age of the micas. However, it was found on further study, that the micas in which the halos were found could be colored or bleached out not only by the alpha particles themselves, but also by the action of heat or perhaps light. In the method which was worked out for the calculation of age from pleochroic halos, it was necessary to know the amount of radioactive material at the center and to find experimentally how many alpha particles were necessary to produce a coloration of intensity equal to that in the mica. However, it is evident that it is necessary to estimate the amount of radioactive material present at the center. Also this method does not take into account the fact that the substance in which the halos are found has, in all

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probability, been exposed to conditions which might change the coloration in either direction. Therefore, this method cannot be used for very accurate age determinations. 1

The second method of determining age from radioactive disintegration data is the helium method
which is based on the fact that helium is one of the
stable products of radioactive disintegration. Unfortunately, helium is lost from minerals as soon
as they are exposed to the atmosphere, and furthermore, still more is lost during the grinding process
while the mineral is being prepared for analysis.

Also heat will drive off helium and promote diffusion.
Therefore, when the minerals are analyzed, they
always contain less helium than has been generated
in them during their whole life. Thus it becomes
evident that any age calculated from this data will
give a minimum estimate.²

The third and probably the best method depending on radioactive data is the lead method. In this

- 1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
 Holmes, and Brown. Published by "The National
 Research Council" as Bulletin 80. pp. 159 ff.
- 2. Ibid pp. 104 ff.

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2. Thid pp. 104 ff.

method the mineral is analyzed in much the same way as in the helium method except that the amount of lead is determined rather than the amount of helium. This method involves the fewest number of assumptions of any of the methods discussed so far, and for that reason, probably gives the most accurate estimate of the age of the mineral.

.

THE LEAD METHOD OF DETERMINING THE AGE OF RADIOACTIVE MINERALS

Boltwood is really the pioneer in this method of determining the age of minerals. He was among the first to offer experimental proof of the disintegration theory of radioactive elements which had been proposed by Rutherford and Soddy in 1903. As Boltwood did his work with the radioactive minerals, he was impressed with the fact that the ratio of the amount of lead and uranium present was not always the same. However, since he found lead present in all the minerals, he came to the conclusion that lead must be one of the final disintegration products of the radioactive elements present in the mineral. Accordingly, he arranged the minerals according to the lead uranium ratio and pointed out the fact that a higher ratio corresponded to an older mineral. In order to be able to express the age of the mineral in years, Boltwood assumed that all the lead present was the result of the disintegration of uranium. Since the time of Boltwood, it has been found that in general his conclusions were correct, but certain corrections must be made for the different isotopes

of descripting the age of minerals. We have brong baen proposed by Rullerford and Boddy in 1905. As the same. However, since as found lead present in sail no hulomos and on amas an ,alaraniz ant lia of the Indianotive elements present in the mineral, of guilfunds alerania out baguarta an , tignibrouch a higher ratio na na behangastron cidar mangid a threat book and ils your dealers booking, Break at was the result of one divintegration of practua. Since the time of volumend, it has been found that in general his conclusions were correct, but certain

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of lead.1

It has been said that lead and helium are stable end products of radioactive disintegration. It is now of interest to see how they are produced by the radioactive element. When a radioactive substance disintegrates, alpha and beta particles and gamma rays are emitted. The gamma rays are electromagnetic waves of very short wave length, and always accompany a beta particle which, in turn, is, in reality, an electron with a velocity very nearly that of light. The alpha particles on the other hand are positively charged particles, also of very high velocity, and with a mass nearly equal to the mass of the helium atom. Actually it has been found that alpha particles are charged helium nuclei. Thus it becomes evident how the helium is formed in the mineral, since an alpha particle becomes an atom of helium merely by picking up an electron.

Next let us see how the lead is produced. It must be remembered that both the alpha and beta

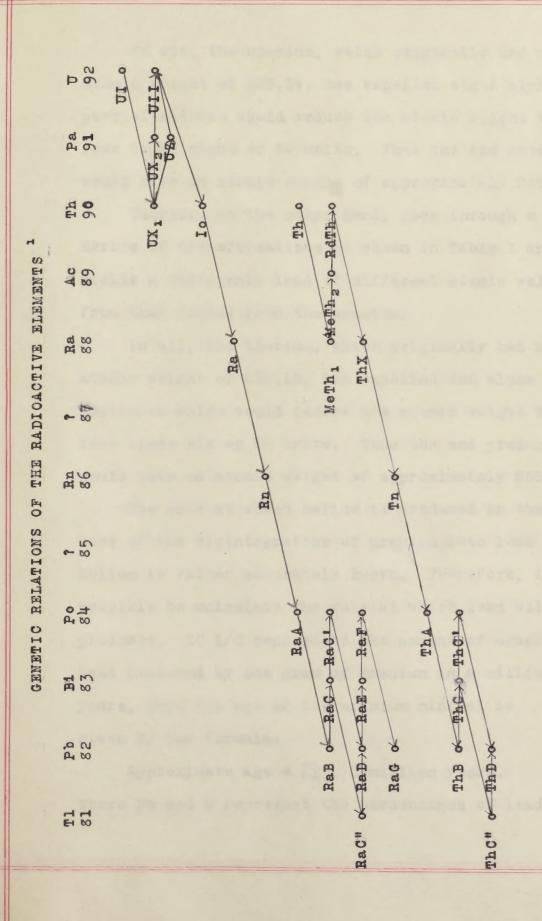
1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
Research Council" as Bulletin 80. p. 73

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particles come from the nucleus of the parent element. Thus, when an alpha particle is expelled, the excess positive charge on the nucleus is decreased by two, and the resulting element would fall two places lower in the periodic table than its parent. However, when a beta particle is expelled, the excess positive charge on the nucleus is increased by one and the resulting element would fall one place higher in the periodic table.

Thus let us start with uranium I which first expels an alpha particle with the formation of uranium X_1 which is two places lower in the periodic table. The uranium X_1 in turn expels a beta particle with the formation of uranium X_{11} which is one place higher in the periodic table. Since the alpha particle has a mass nearly equal to four, the uranium X_1 formed has an atomic weight approximately four less than the atomic weight of uranium I. However, since the mass of the beta particle is practically zero, the uranium X_{11} has the same atomic weight as the uranium X_1 from which it was formed. Uranium continues to disintegrate in a manner which Table I will make clear and yields a radiogenic lead, RaG, with an atomic weight of 206.

Cruss lot no start with account young first expels on elpha periods with the formation of arenium \$\frac{1}{2}\$ which is two places lover in the periodic table. The utenium \$\frac{1}{2}\$ in ours expels a beta partiture with the formation of arenium \$\frac{1}{2}\$ which is one piece higher in the periodic table. Since the clapse uranium \$\frac{1}{2}\$ formed has a desa nearly equal to four, the arenium \$\frac{1}{2}\$ formed has an about weakly approximately four lets than the slowle weight of utenium \$1\$. Nowers, since the mess of the best particle is practically sero, the utenium \$\frac{1}{2}\$ from which it was formed. Utenium continues to disintegrate in a menner which Teins Teins \$1\$ with an atomic weight of 200.



"An Outline of Atomic Physics" Physics Staff of the University of Pittsburgh.

In all, the uranium, which originally had an atomic weight of 238.14, has expelled eight alpha particles which would reduce the atomic weight by four times eight or 32 units. Thus the end product would have an atomic weight of approximately 206.

Thorium, on the other hand, goes through a series of transformations as shown in Table I and yields a radiogenic lead of different atomic weight from that formed from the uranium.

In all, the thorium, which originally had an atomic weight of 232.12, has expelled six alpha particles which would reduce the atomic weight by four times six or 24 units. Thus the end product would have an atomic weight of approximately 208.

The rate at which helium is produced in the case of the disintegration of uranium into lead and helium is rather accurately known. Therefore, it is possible to calculate the rate at which lead will be produced. If 1/C represents the amount of uranium-lead produced by one gram of uranium in a million years, then the age of the uranium mineral is given by the formula:

Approximate age = $\frac{Pb}{U}$. C million years. Where Pb and U represent the percentages of lead

In all, the arentum, which originally has an acomic value of 200.14, and deposited eight alpha atomic value of 200.14, and deposite atomic value of 200.14 and a the atomic value of 200.15 and 12. Thus whe ent product would have an atomic weight of approximately 200.

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Lead produced by one gram of granium is a million forms, then the gram of granium is a million given by the formula:

Approximate age = 20 . O million years.

and uranium respectively in the mineral. The formula assumes, of course, that the mineral was originally lead free and that no loss or gain of lead from external sources has taken place since the formation of it.

Since most of the uranium minerals also contain thorium, it is necessary to take this thorium into account. In order to do this, the amount of uranium equivalent in lead-producing power to one gram of thorium is determined. This value is then represented by k. The age of a pure and uncontaminated thorium mineral would then be given by the formula:

Approximate age = $\frac{Pb}{k.Th}$. C million years. In the case where both uranium and thorium are present, the time required for the production of all the lead is given approximately by the combined formula:

Approximate age = $\frac{Pb}{U+kTh}$. C million years.

Using $1.52 \times 10^{-10} \text{yr-1}$ as the most acceptable value for the disintegration constant of uranium, the value of C has been calculated to be 7,610, but the adopted value has been rounded off to 7,600.

The value of k has also been calculated by the following method: Equal amounts of uranium and

and urani m respectively in the mineral. The formula assumes, of course, that the hineral was formula assumes, of course, that the lone or gain of lead from external sources and taken place since the formation of it.

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thorium respectively will produce amounts of lead proportional to

$$\lambda_{\mu} \frac{206}{238}$$
 and $\lambda_{\pi} \frac{208}{232}$

Therefore, by definition

$$k \cdot \lambda_{u} \frac{206}{238} = \lambda_{\overline{u}} \frac{208}{232}$$

$$k = 1.035 \frac{\lambda_{\overline{u}}}{\lambda_{u}}$$

$$\lambda_{\overline{u}} = 5.33 \ 10^{-11} \text{yr}^{-1}$$

$$\lambda_{u} = 1.52 \ 10^{-10} \ \text{yr}^{-1}$$

$$k = 1.035 \frac{5.33 \ 10^{-11}}{1.52 \ 10^{-10}}$$

$$k = 0.36$$

Using these values for C and k the age formula becomes

Approximate age Pb U+.36Th . 7,600 million years. It is interesting to consider next some of the advantages and difficulties involved in the lead method of determining the age of minerals. In the first place there are three distinct kinds of data which must be rather accurately determined for any method of age determination.

1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
Research Council" as Bulletin 80. pp.199 ff.

thorium respectively will produce amounts of lead

A 208 A BOS A

Therefore, by definition

808 / = 808 /. x

M_880.I = X

N= 0,55 10-11/31-1

A= 1.88 10-10 yg-1

11- 01 86.8 860.1 = X

35.0 = X

Using these values for C and E the age Furnal:

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1. "The Age of the March" Hompp, Schwonert, Meveril, Edimes, and Brown. Published by "The Hational Research Council" as Fulletin 80, pp.199 ff.

- 1. The rate at which some selected process is going on at the present time must first be determined.
- 2. The total change which has been caused by this selected process during the interval since it first began must be measured.
- 3. The law of variation in the rate of the selected process over the duration of the interval it is hoped to measure must also be shown.

This last condition is often overlooked, but that makes it no less important. In many cases present day rates are altogether different from the rates which have prevailed throughout the time it is hoped to measure.

It is interesting to note here that none of the first three methods of age determination given at the beginning of this paper meet these requirements, so they are, therefore, of more historical interest than value. However, let us now turn to the radioactive methods of age determination, and especially the lead method.

1. In the case of uranium the present day rates of disintegration are known with a fair degree of accuracy. Also those of thorium are known approximately.

- 1. The rate at which some selected process is soing on at the present time must first be determined.
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1. In the case of wrinium the present day rates of district and with a fair degree of accuracy. Also those of thorium are known approximately.

- 2. The total change since the formation of suitable radioactive minerals of known geologic age can be determined by chemical analysis accompanied by atomic weight determinations. Here "suitable radioactive minerals" means (a) that the minerals are unaltered by external influences since the time of their crystallization so that it may be assumed safely that no lead, uranium, or thorium have either been removed or introduced into the mineral; and (b) that the minerals were either initially free from lead, or, if such lead were present, it may be detected by atomic weight determinations, and its presence allowed for in the calculations. In the case of the helium method, the second condition is not met perfectly since all the helium produced during the interval is not retained by the mineral. It is lost especially while the mineral is being prepared for analysis, and for this reason ages given by the helium method are, at best, a minimum figure.
- 3. The rate of production of lead isotopes by radioactive elements during the whole of geologic time is believed, with ever increasing confidence, to be constant. That is, it is in complete agreement

R. The total oh mre since the formation of by atomic weight determinentons. Here "saitable safely that no lead, uranium, or thorium have either from lead, or, if such lead were present, it may be descoted by atomic weight determinations, and 17s oresence allowed for in the calculations. In the case of the notion method, and second condition is during the inversal is not retained by the minetal. prepared for analysis, and for this reason ages given by the halled method are, at best, a minimum figure.

5. The rate of production of lead isotopus of radiosotive elements during the whole of yeologic time is believed, with ever increasing confidence, to be constant. That is, it is in complete agreement

with the disintegration theory of Rutherford and Soddy, and does not vary in any way whatsoever.

There has been a great deal of experimental work done in an attempt to see if the most drastic of physical conditions would have any effect on the rate of radioactive disintegration. The radioactive elements have been subjected to temperatures ranging from that of liquid air to 2,500°C, and it was found that the law of disintegration held throughout the whole range. Very high pressures or a strong magnetic field also failed to change the quality or quantity of the radiations. It does not even matter whether or not the radioactive element is in solution, in chemical combination, taking part in chemical reaction, or suffering bombardment by X, alpha, beta, or gamma rays since, in all cases, the disintegration goes on as usual. This convincing experimental proof is the cause of the belief that the rate of the production of lead isotopes has not changed throughout geologic time. -

1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
Research Council" as Bulletin 80. p. 145

Soddy, and does not vary in any way whatevers. rate of radiosctive disintegration. The radioont the whole runge. Very high pressures of a strong ration nevs lon seed il .ano Halber and to vilinaup reaction, or suffering conbardment by X, alpha, bets, or gamma rays since, in all cases, the disintegration goes on as usual. This convincing not changed throughout geologic time. 1

1. "The Age of the Carth" Mnapp, Schwohert, Kevarik, Holmes, and Brown. Published by "The Hattonkl Research Council" as Bulletin 60. p. 145

Now let us turn to the things we must know in order to have a solvable problem. The first and possibly the most important thing to know is that the mineral with which we are working has not been changed either by taking up material from the outside, or by losing to the outside some of what was originally present in it. In addition we must know the following things:

- 1. The data from the analysis of the mineral, that is, the masses of uranium, thorium, and lead (all isotopes) per given mass of mineral.
- 2. Atomic weight of the lead (all isotopes) from the same material.
- 3. The disintegration constants of all the radioactive elements involved.
- 4. The atomic weights of the different lead isotopes separately.
- 5. The parental lineage of the various isotopes of lead.
- 6. The possibility of the presence of common lead at the time of formation of the mineral.

Now let us consider these requirements in a little more detail. In the case of 6, it is possible to allow for the presence of common lead in the

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calculations and therefore this requirement should cause no trouble. As for 3, 4, and 5, these have all been fairly accurately determined. 1 and 2 should be determined by the analysis. This leaves for consideration only the possibility of alteration since the time of formation.

In the ordinary analysis, care was taken to select what appeared to be an unaltered crystal.

In some cases, the very outermost layer might have been removed before analysis. The rest of the crystal would then be ground up and the analysis carried out on it. This procedure assumed that no leaching had occurred or, if it had, it had gone no further than the outermost layer.

Arthur Holmes points out that, in spite of their superficial appearance of freshness, many thorium minerals have been altered. To account for this, Holmes gives a selective leaching hypothesis. According to this hypothesis, the thorium lead is leached out faster than uranium lead. When an atom

1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
Research Council" as Bulletin 80. pp. 213 ff.

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1. "The Age of the Ratth" Finapp, Schlichert, Keverik, Holmes, and Brown. Published by "The Mational Heaserch Council" as Bulletin 80. pp. 215 ff.

of lead comes into existence in a crystal of uraninite, it is surrounded by a lattice of uranium and oxygen atoms which make up an acidic medium. At first the arrangement would not be at all regular, but as water passed slowly through the crystal, the mobility of the atoms would be increased and it seems probable that the most insoluble compound would be formed, namely lead uranate. This would prevent more lead from being lost. However, when an atom of lead comes into existence in a crystal of thorite, it is surrounded by thorium and oxygen atoms which do not create an acidic medium, and therefore, there is no such compound as lead thorate. As a result, the lead exists in a relatively soluble form, that is the oxide.

The following table gives the results of some analyses of thorium minerals:

Ref. No.	Minerals from Ceylon	Percent	ages of Th	Pb	Lead Ratios
1	Thorite	0.72	54.45	0.36	.019
2	Thorite	3.50	59.2	0.78	.031
3	Thorite	4.57	62.8	1.28	.047
4	Thorite	1.87	65.3	1.71	.067
5	Thorianite	11.8	68.9	2.34	.064
6	Thorianite	20.2	62.7	3.11	.073
7	Thorianite	26.8	57.0	3.50	.074

of lead comes into existence in a crystal of arcanite, it is surrounded by a lettice of uranium and oxygen aroms which make up an actife medium. At first the arrangement would not be at all regular, but as water passed alowly through one orygetal, the moullity of the atoms would be increased and it neems probable that the most inspisable compount would be formed, namely lead arenate. This would prevent more lead from being lost. However, when an atom of lead ecrasting oxistence in a crystal of tenrite, it is surrounded by therein and crystal of tenrite, it is surrounded by thereing and crystal of tenrites, it is surrounded by thereing, and therefore, there is no create an acidic medium, and therefore, there is no much compound as load shorate. As a result, the lead exists in a relatively solucie form, that is the oxide.

The following paule gives the results of some

				5
910.	0,36	34,10	311	
.081	87.0	4	08.8	
740.	1,26	8,88	4.57	
780.	1.71	8.30	1,87	
180.	A8,8	0,88	0.11	
.078	2.11	62.7	5,05	
170,	08.8	0.78	16,88	

A pitchblende from Ceylon which belonged with this same set of minerals gave a ratio of .065. Supposedly all the ratios should be alike if the thorites and thorianites have remained unaltered. The very low result for the thorites indicate that the lead has been leached out of those minerals. 1

Thus it seems that in the case of mixed minerals such as the thorianites rich in uranium, the tendency to lose lead is less than in the minerals poor in uranium. This follows from the fact that the thorium-lead on its way out of the uranium rich mineral must pass through an assemblage of uranium and oxygen atoms. Thus part of the lead which would otherwise escape is fixed as lead uranate.

Thus it is evident that in the case of uraninite it is necessary to have a perfect crystal. In the case of age determinations of thorium minerals, it is better to take those with a high percentage of uranium since this has a tendency to prevent the loss of lead by leaching.

1. "The Age of the Earth" Knapp, Schuchert, Kevarik,
Holmes, and Brown. Published by "The National
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1. "The Age of the Berth" Knapp, Sandomart, Heyarik,
Tolmes, and Brown. Published by "The Madional
Research Council" as Bulletin 80. p. 214

THE WILBERFORCE URANINITE

There are several minerals which contain both lead and uranium and in some cases thorium also. The percentages of the three elements varies considerably from one mineral to another, but the lead-uranium ratio is fairly constant for minerals known to be of approximately the same geologic age. Of these minerals, uraninite is perhaps the best to work with in age determinations, because first, it occurs in a relatively pure state, and second, it occurs in a rather definite crystal form. Another distinct advantage is that there is enough of the uranium, lead, and thorium present to give a satisfactory analysis.

World. Among other places it occurs in the
Wilberforce region of Ontario, Canada. The first
discovery of radioactive minerals in the Wilberforce
region was made by Mr. W. M. Richardson in 1922.
Mr. Richardson was a prospector and miner with
experience in Alaska. He took up residence, however, in Cardiff township, about one-half mile northwest of the present mines. In the course of his
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as far as is known, is the only radioactive mineral occurring in any important amount on his property. 1

The uraninite occurs in crystals or crystal masses imbedded in feldspar. It may also occur in the feldspar lining of a calcite-fluoite area, or as an irregular mass in magnetite. When the uraninite occurs in this last way, it shows signs of being considerably altered. The freshest mineral is that which is entirely embedded in feldspar. The size of the uraninite crystals ranges from about one-half inch diameter to about two inches across. They are predominantly cubic, but are sometimes modified somewhat by the octrahedron. The faces are often pitted or indented and perfect crystals are rare. When the uraninite occurs as irregular masses, these may range from the size of a pea to several pounds.

It has been pointed out that the uraninite which occurs within twenty feet of the surface

1. "The Wilberforce Radium Occurrence" H. S. Spence and R. K. Carnochan. Canadian Mining and Metal-lurgical Bulletin, being a part of the Transactions of the Canadian Institute of Mining and Metallurgy for 1930. p. 6

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usually appears to be considerably altered. Also, this alteration often penetrates to a considerable depth in the crystal itself, and in some cases, the entire crystal seems to have suffered from alteration. The work done by Alter and Kipp indicates that, even in the case of what appeared to be a fresh crystal, alteration had penetrated at least through the first third of the crystal. 1

Both Ellsworth and Todd have analyzed the Wilberforce uraninite. Their results are given in the following table:

Analyst	%Lead	%Uranium	%Thorium	Lead-Ratio
Todd	9.65	60.56	10.02	.150
Ellsworth	10.25	55.26	11.92	.171
Ellsworth	10.19	61.44	9.32	.157

The first is by Todd, and the last two by
Ellsworth. The first analysis was done on probably
the freshest material of the three. The second, or
the first done by Ellsworth, was done on uraninite
which was known to be considerably altered. The

1. "Pegmatite Minerals of Ontario and Quebec"

H. S. Spence. American Mineralogist 15 Nos. 9-10

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third was done on a crystal which was apparently fresh, and it is seen that the results of this analysis agree fairly well with that done by Todd.

It is interesting to note that the best Wilberforce minerals yield ratios of .150 and .157 which are in agreement with other uraninites of the same geologic period from Ontario. Also as a usual thing, minerals high in thorium give a ratio inconsistent with those of low thorium content, but the results from this mineral which contains about ten per cent thorium agree with others from an Ontario mineral with much lower thorium content.

As has been pointed out earlier in this thesis, the work done on the determination of the age of the earth by means of the lead-uranium ratio has, in general, assumed that no alteration has taken place, or that if there were any alteration, it would only penetrate a very short distance into the crystal. However, actually, in many cases it has been shown that alteration has penetrated to a considerable depth and has sometimes gone throughout the crystal.

In general in the analysis of the crystal, it has been either ground up and used without removing

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In general in the analysis of the orystal, it

any of the outside, or a little of the outside has been taken off and then the rest of the crystal used. By using this method, the workers have assumed either that there has been no leaching, or if any leaching has occurred, all three elements have been leached out in the same proportion. If both of these assumptions were known to be perfectly true, the leaduranium ratios would be trustworthy in the age determinations. However, if leaching does occur, as it does in all probability, and all three elements are not leached out in the same proportion, the leaduranium ratios are worthless.

The two analyses by Ellsworth and the one by Todd serve to illustrate this point very well. The crystal which appeared altered and was analyzed by Ellsworth gave a lead-uranium ratio of .171 which corresponds to an age of 1,300 million years. The other crystal done by Ellsworth which appeared perfectly fresh gave a ratio of .157 which corresponds to an age of 1,190 million years. The crystal done by Todd which also appeared perfectly fresh gave a ratio of .150 which corresponds to 1,140 million years. Thus it is seen that the analyses of the fresh mineral check fairly well, while that

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done on the altered mineral gives a high ratio and a high age.

The high ratio obtained on the altered mineral might indicate that the uranium had been leached out in preference to the lead which would result in a higher ratio since the uranium content would thus be diminished in relation to that of lead and thorium. It is also conceivable that the lead might be leached out in preference to the uranium and thorium which would of course, result in a lower ratio. What is probably the case, is that all three of the elements are leached out, but some are taken out to a greater extent than others.

There had been no attempt to bring any quantitative data to bear on this subject until Alter and Kipp did their work last year. In this work the crystal which was used was a specimen of Wilberforce uraninite. It was apparently unaltered, cubic, and possessed definite cleavage faces. It weighed about twenty-four grams and was about three-quarters inch in each dimension. The procedure used was as follows:

The crystal was carefully dried and weighed.

It was then placed in dilute nitric acid and left

done ou the altered mineral gives a high ratio and a high ship age.

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It was then removed with a pair of glass tongs and was carefully rinsed into the solution. It was then dried and weighed. This gave the first layer. The procedure was repeated to remove a second layer, and the rest of the crystal was used as the third layer or core. It was pointed out in this work that as soon as the crystal was put in acid, it became evident that it was not a perfectly homogeneous crystal since the previously smooth faces became pitted by the acid showing that the acid was dissolving out some places faster than others. However, the core appeared as a pure black substance. The results obtained are as follows:-

OUTSIDE LAYER

				Average	
Lead	9.92%	9.32%		9.63%	
Uranium	38.11%	37.73%	37.74%	37.86%	
Thorium	8.38%	8.34%		8.36%	
Lead-Uranium Ratio .235					
Age 1,846 million years					

there until about one third and apparently discolved. It was then removed with a pair of glass tough and was carefully rineed into the solution. It was then dried and weighed. Into gave the first layer. The procedure was repeated to remove a second layer, and one rest of the crystal was need as the third layer or core. It was pointed out in this work that as moon as the crystal was put in sold, it became evident that it was not a perfectly homogeneous crystal since the previously shooth faces became pitted by the sold showing that the sold was dissolving out some places factor and others. Now-solving out some places factor and others. Now-see, the core appeared as a pure bluck substance.

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Lead 2.325 2.325 9.535
Uranium 35.115 37.735 37.745 37.345
Thorium 8.385 8.34 8.345
Lead-Branium Batio .235

Age 1,606 million years

MIDDLE LAYER

			de Communa	Average	
Lead	11.91%	11.97%	11.95%	11.94%	
Uranium	58.51%	58.57%	58.48%	58.52%	
Thorium	14.13%	14.07%		14.10%	
Lead-Uranium Ratio .188					
Age 1,426 million years					

CORE

			Average
Lead	11.89%	11.87%	11.88%
Uranium	60.71%	60.62%	60.67%
Thorium	8.10%	8.02%	8.06%

Lead-Uranium Ratio .187

Age 1,420 million years

ENTIRE CRYSTAL

Lead 11.09%

Uranium 51.89%

Thorium 9.97%

Lead-Uranium Ratio .199

Age 1,512 million years1

1. "The Effect of Leaching on the Lead-Uranium-Thorium Ratio of a Crystal of Uraninite" E. M. Kipp.

Thesis 1935 Boston University Graduate School,pp.23-4 or Science 82, 464, 1935.

HEYAI SAGUEN

AVEREGE

TAG.II MEG.II MEG.II MEGI

Uranium 58.51 56.57 58.65 58.524

Thorium 14.15 14.078 14.10%

Lead-Uranium Ratio .183

Age 1,426 million years

CORDS

AVELEES

lead 11.89% 11.89% 11.88%

Branium 50.71% 80.62% 60.675

Thorium 8.105 8.025 multionT

Lead-Urenium Patio . 187

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1. "The iffect of Leaciding on the Lead-Urani ma-Thorium Ratio of a Grystal of Braninite" I. M. Ripp.
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The results indicate that leaching does occur, but that a fairly accurate age determination can be obtained by removing the first third or more of a crystal before analysis since the ratios obtained for the inner two layers were practically identical.

Objections have been raised to the above procedure since it is feared that the acid effected some preferential leaching of its own which would give erroneous results as far as the leaching done by nature is concerned. Accordingly, the present work has been started in which the crystal has been divided into three portions mechanically so that there could be no danger of preferential leaching in the analysis itself.

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EXPERIMENTAL PROCEDURE

of the crystal so that no leaching could be attributed to the action of the acid, a variation in the
lead-uranium ratios between the inner and outer
layers of a crystal would indicate that leaching
had taken place somewhere in the crystal. On the
other hand, if no variation were found in the ratios,
this would indicate, possibly, that there had been
no leaching in that particular crystal.

The crystal used in this work was a rather good specimen of Wilberforce uraninite. It was predominately cubic in form although it looked as if it had grown against something so that one half of it had not had a chance to develop. On this side which appeared to have been against some other substance, there was a small round spot of a brownish material. This spot persisted all the way through the second layer as it was taken off. The rest of the crystal did not appear to be altered at all and was a good, dark greenish black in color. The whole crystal weighed about thirty-one grams.

MATINETONS LANDOUTS, SEE

If sold were not used to separate the layers
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STANDARDIZATION OF WEIGHTS

Although a new set of weights was being used for this work, it was thought necessary to standardize them before use. In order to do this, a method of substitution was used, and the weights could then be weighed wholly on one side of the balance. This method of substitution has two advantages: (1) it eliminates the effect of any inequality in the length of the arms of the balance, (2) it awoids mental confusion resulting from the continual interchanging of weights from one pan to the other as is done in other methods.

In the set of weights to be standardized, all the fractional weights were of platinum, and taken together, were equivalent to one gram. The rest of the set were made of gold plated brass. The different weights of the same denomination were all marked in some way which would serve to distinguish between them.

The zero point of the empty balance was determined. One of the centigram weights was placed on
the left-hand scale pan. A tare centigram weight
from another box was then placed on the right-hand
pan. A five milligram weight was also placed on the

CTELLEN TO BOTTARIONAMIATE

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right-hand pan in order that the rider might fall somewhere near the center of its path. The swings of the balance with this centigram load were then carefully recorded. The rider was then moved one milligram, and the rest point of the balance again determined. From this the sensibility of the balance was calculated by the following formula.

Sensibility = .001/displacement of rest point by addition of 1 mg. wt. From the sensibility, the true rider was found by multiplying the sensibility by the difference between the rest point (before the addition of the one milligram weight) and the zero point of the empty balance. The sign of this correction of course is determined by which way the rest point is displaced from the zero point.

After this had been done, the first centigram weight was replaced on the left-hand pan by a second centigram weight. The new rider reading was determined exactly as before. The difference in the two, of ourse, gives the difference in the weight of the two centigram weights. The second centigram weight was then replaced by the third and the process repeated.

right- and sen to order that the rider might feld somewhere near the center of the pash. The swinge of the columns with this centisted load were then earefully recorded. The rider was then moved one milligron, and the rest point of the behance again determined. From this the sensibility of the halance was calculated by the following formula.

Sensibility = .oul/Haplacedent of rest point of medition of 1 ag. wt. From the mensimility, the statistic that the statistic the statistic the statistic that the statistic of the engry belance. The sign of this cut-rection of course is determined by which may the rest point is displaced from the state point.

After this had ocen done, the first contigran
wellah was replaced on the left-hand pan by a second
centilerum watghe. The new rider reading was datermined amostly as before. The difference in the two,
of course, gives the difference in the weight of
the two dentigran woisnes. The second centigram
weight was then replaced by the third and the process
repasted.

The first and second centigram weights were then placed on the left-hand pan, and another tare centigram weight on the right-hand pan. The rider was adjusted, and the rest point, sensibility, and true rider found as before. The two centigram weights were then replaced by a two-centigram weight and the process repeated.

By following this procedure, every weight was compared with every other of the same denomination and with the combination of all of the smaller weights. From the data thus obtained, it is possible to calculate the weight corrections by a method which the following table and explanation will make clear.

The first and second centifron weights were than placed on the left-hand pan, and another tare centifren weight on the right-hand pan. The rider was adjusted, and the rest point, sensibility, and true rider found as before. The two centifran weight weights were then replaced by a two-centifran weight and the process repeated.

Dy following this procedure, every weight has compared with every other of the same demonination and with the compination of all of the samilton weights. From the data time obtained, it is possible to calculate the reight corrections by a method which the following table and explanation will make clear.

Left Pan	Rider	Rider Differ- ences	Preliminary values (actual)	Aliquot parts of 10.00738 (ideal)	Corrections Actual minus ideal
g.	mg.	mg.	€.	g.	mg.
.01' .01" .01 .01" .01" .01"	4.90 4.91 4.83 4.93	+.01 07	.01000 .01001 .00993	.01001 .01001 .01001	01 .00 08
.02	4.97	+.04	.01998	.02001	03
.02+01'	5.06	+.12	.05004	.05004	00
.05+01'	4.94 4.97 5.06	+.03 +.12	.09999	.10007	08 +.01
.10'+.10 .20 .20+01'	5.05 5.12 4.87	+.07	.20014	.20015	01
.50 .50+01'	5.03	+.16	.50033	.50037	+.04
1.0	5.31	4.38	1.00088	1.00073	+.15
2.0"	5.46 5.30	+.45	2.00183	2.00148 2.00148	+.35 +.19
2.0"+1.0	7.89 7.21 5.98	68	5.00370	5.00369	+.01
5.0+1.0 10.0" 10.0'	5.08 5.28	90 70	10.00718 10.00738	10.00738 10.00738	20
10"+10'	.40	20	20.01436	20.01476	40

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	10.00Vad				
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10	TOOLO.	00010.		oe, A	1.00.
00.	ICOLO.	LOOLO.	10.4	Di.	F.00.
80,-	TOOLG.	S4800.	70,-	4,83	IO.
				60.	10.4"10.
50,-	.02001	09010	40.4	70.4	80.
				100	*10+HO.
00	400E0.	.05000	SI.4	80.8	. 05
				ANA	10,100.
08	10000	00001.	+,03 +,12	79.4	101,
20.0	. TO000A	\$000I.	SI.	80.6	01.
				00,0	01, +101,
111,-	d_E008'	ALCOS.	4.07	81,8	02,
				- 88.5	110,+08.
10.4	78008.	.59083	7.16	00,0	1000.
4.15	1.000073			A =	, TO * 609 *
	arono.T	1,00038	86.4	10.0	0.1
4.88	2,00143			10.0	1.0,+0,1
8.0.4	2,00248	8.00169 8.00169	28.4	80.0	10.3
		10,000.0	200	05,5	*0,8
10.+	5.00369	0,000,0		78,5	0.140.8
		•	88	I.CI	0,3
08	10.00758	areco.or	00	D. 90	0,1+0,1
,	10.00/55	10.00730		80.8	10.01
		******	01 = ==	88.0	10.01
04	80.01476	20.01A36	05	04:	

The "preliminary values (actual) are found by assuming that the first centigram weight actually weighs one centigram. The rider differences for the next two centigram weights are then applied to .01 gm. In order to find the preliminary value for the .02 gm. weight, the preliminary values for the two centigram weights used in the procedure are added, and the rider difference for the two-centigram weight applied to this figure. For the five-centigram weight, all the foregoing preliminary values are added together and the rider difference for the five-centigram weight applied to this figure. This process is repeated throughout the table. The rest of the table is self explanatory.

The "preliminary values (social) are found by assuming that the first centifrem weight socially weighs one centifrem. The rider differences for the next two centifrem weights are then spolied to .01 cm. next two centifrem weights are then spolied to .02 cm. In order to find the preliminary values for the two centifrem weights used in the procedure are added, and the rider difference for the two-centifrem weight applied to this figure. For the two-centifrem weight, all the foregoing preliminary values are added together and the rider difference for the five-centifrem weight, all weight applied to this figure. This process is repetited to this figure. This process is repetited to this figure. The rest of the table is self explanatory.

METHOD OF WEIGHING

In weighing, the direct method was used throughout. The zero point of the empty balance was first determined. The crucibles were kept in a desiccator which had concentrated sulfuric acid in it as a dehydrating agent. They were removed from this desiccator and were placed directly on the left-hand pan of the balance which was a Troemner No. 10 balance. The standard weights were placed on the right-hand pan, the rider was adjusted, and the rest point carefully determined. The sensibility was then determined in the same way as was used in the standardization of weights, and the true rider calculated. The weight corrections were then applied to the various weights, and finally the true corrected weight was found.

A sample notebook page will serve to show the method of weighing. As will be noticed, all the weights were recorded separately in order that the proper weight corrections might be applied immediately or after a considerable lapse of time.

DETROITE NO CONTENT

In weighte, the direct method was used throughout. The zero point of the many calcure was first
determined. The crucibles were kept in a desiccator
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A sample notebook page will serve to show the method of weighing. As will be noticed, all the weights were recorded separately in order that the proper weight corrections might be applied immediately or after a considerable layer of time.

Sample Notebook Page

Zero point of empty balance : .08 1

Pt. crucible No. 4 on left

Average rest point = 2.81 left

Sensibility = $\frac{.001}{2.86}$ = .00035

Rider correction = $-.00035 \times .13 = -.00005$

True rider = -.00355

Weight = 30.08645

Weight corrections for weights used = -.52 mg.

True weight of Pt. crucible No. 4 = 30.08593

I BO. I someLee wigms to Integ ones

Pt. grupiple No. 4 on left

.tr do. = iniog jast systava

8.25 8.7 8.25 2.8 8.7 2.7 3.7 8.76

Jist 18.2 = sping Jest system

Sensipility = 2.00035

Rider correction = -.00000 x .18 = -.00000

True rider = -,00555

Weight = 50.08645

Weight corrections for weights used = -. UR mg.

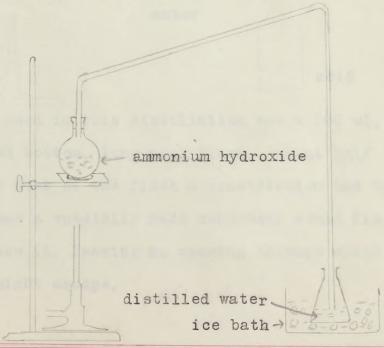
True weight of Pt. crucible No. 4 = 50.08595

PREPARATION OF PURE REAGENTS

Ammonium Acetate. - Baker's C.P. grade ammonium acetate was used. The proper amount was dissolved in distilled water and the solution was filtered before use.

Ammonium Carbonate. - Baker's C.P. grade ammonium carbonate was used, and was dissolved in distilled water or used as a solid.

Ammonium Hydroxide. - The C.P. grade of ammonium hydroxide was used. It was redistilled by placing it in a 500 ml. pyrex flask fitted with an air condenser. The bottom of the condenser almost touched the surface of the distilled water into which the ammonia was being distilled. The apparatus will be made clear by the following drawing.



STEARANT OF PURCH PLANTENTS

Armunium Acctate, - Baker's C.F. grade amnonium acetate was used. The proper amount was discolved in distilled water and the solution was filtered before use.

Amaonium Carbonate. - Baker's C.P. grade amonium carbonate was used, and was displyed in distilled water or used as a solid.

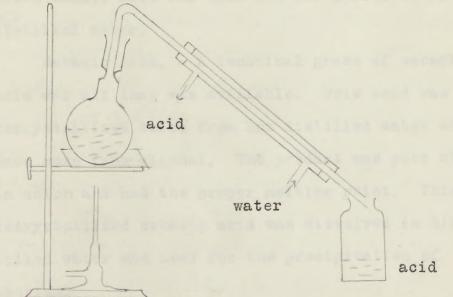
Aumonium Hydroxide, - The C.F. grade of ammonium hydroxide was used. It was redistilled by placing it in a 500 ml. pyrex rises fitted with an air condenser. The untion of the condenser almost touched the surface of the distilled water into which the ammonia was being distilled. The apparatus will be made clear by the following drawing.

ammunitum hydroxide

dani ant

The ammonium hydroxide in the flask was heated to boiling for about fifteen minutes while the ammonia distilled over and was absorbed by the distilled water. This distilled water was kept in an ice bath while the distillation was being carried out.

Hydrochloric Acid. - Baker's C.P. grade
hydrochloric acid was used. However it was distilled
before use. The apparatus in which the distillation
was carried out is shown in the following drawing.



The flask used in this distillation was a 500 ml.

pyrex round bottom, long neck flask. About half

way up the neck of the flask a constriction had been

made so that a specially made condenser would fit

tightly into it, leaving no opening through which

the acid might escape.

The amnonian hydroxide in the flack was heated to boiling for about fifteen minutes while the amonia that distilled over and was absorbed by the distilled water was kept in an ice text while the distillation was being carried out.

Hydrocaloric Acid. - Haker's C.F. grade bydrocaloric acid was used. Wowever it was distilled before use. The apparatus in which the distillation was carried out is shown in the following drawing.

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The fluor was a firtiliation was a for al.

Dyrex round bottom, long near flash, About half

way up the meak of the flash a constriction and been

made so that a specially made condenser would fit

tightly into it, leaving no opening through which

the acid might escape.

The acid was diluted 1:1 and was placed in this flask and was distilled in the usual way, the first and last thirds being discarded.

Nitric Acid. - Baker's C.P. grade nitric acid was used. The concentrated acid was redistilled in the same apparatus as was used for the hydrochloric acid. It was not diluted, but was used directly.

Oxalic Acid. - Baker's C.P. grade of crystallized oxalic acid was used and was dissolved in distilled water.

Sebacic Acid. - A technical grade of sebacic acid was all that was available. This acid was recrystallized twice from hot distilled water and once more from alcohol. The product was pure white in color and had the proper melting point. This recrystallized sebacic acid was dissolved in distilled water and used for the precipitation of thorium.

Sulfuric Acid. - Baker's C.P. grade of sulfuric acid was used. It was redistilled using a pyrex retort in which to carry out the distillation. A ring burner was used to furnish the heat, and was applied directly to the retort.

The soid was diluted led and was placed in this flast said was distilled in the dated way, the first end last thirds being discarded.

With Acid. - Haker's U.T. grade mitrio sold was used. The doncentrated acid was redicilled in the same apparatus as was used for the hydrochloric acid. It as not diluted, but was used directly.

Omalio Acid. - Haker's U.T. grade of crystal-lined oxalic acid was used and was dissolved in distilled water.

Senate Acid. - A technical grade of debacto acid was all that was available. This sold was recrystallized twice from hot distilled water and once more from alcohol. The product was pure widte in color and had the proper multine soint. This recrystallized sepacto acid was dissolved in that the precipitation of therium.

Sulfurio Acid. - Saker's C.F. grade of sulfurio acid was used. It was redistilled using a pyrex recort in which to carry out the distillation. A ring barner was used to furnish the heat, and was spalled directly to the recort.

DIVISION OF CRYSTAL INTO LAYERS

At first it was hoped that a lapidary might be found who would have instruments such that he would be able to cut the crystal into the proper layers. However, there seemed to be none with instruments suited to so large a crystal.

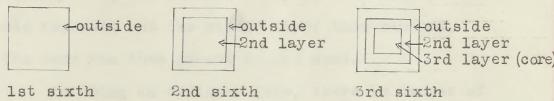
The next thing which was tried was cutting the crystal with a diamond saw. Since our laboratory did not have a diamond saw, Prof. L. C. Graton, Prof. Charles Palache, and Mr. Harry Berman, all of the Mineralogical Laboratories of Harvard University, were consulted. However, they pointed out that it would be an almost endless task to cut each face off separately in such a way as to divide the crystal into three distinct layers. They did think it might be possible to seal the crystal in bakelite and then by means of a few cuts divide it so that it might be used. The general method was as follows: - The crystal would be sealed in bakelite and the cuts made parallel to each other and parallel to one face of the crystal so that the crystal would be divided about into sixths. If this were done accurately, the first sixth would consist practically entirely of the outermost layer of the crystal, the

SERVAL DIVIS TATESAND AD MOISTAID

At first it was noted that a lapidary might he found who would have instruments ench that he would be able to out the crystal into the proper layers. Here seemed to be more with instruments suited to so large a crystal.

orystel with a dramond cam. Since our laboratory and have a dismond saw, Prof. L. C. Craton, Prof. Charles palache, and in. Harry Berman, all University, worde consulted, However, they pointed the crystal into three distinct layers. They did saw honism foreneg and . here ed draim it tand on snob star sing 11 .eddxis ofni soons bebivib ed accurately, the first sixth would consist practically

second sixth would contain a ring of the outermost layer around a core of the second layer, and the third sixth would consist of rings of all three layers. A few drawings will make this separation clear.



This work was actually done under the supervision of the above named members of the Harvard
staff. The crystal used in this attempt was very
kindly furnished us for the purpose by Prof. T. L.
Walker, Director of the Royal Ontario Museum of
Mineralogy. When it was completed, it did not seem
accurate enough for the present work in that the
crystal seemed to be tipped a little so that the
cuts had not been made parallel to one of the faces
of the crystal.

Accordingly, as a final resort, the layers were filed off with an ordinary file. The crystal used in this method is the one described above. It had been kept carefully wrapped, and was simply taken and put in a clamp and filed. Marks were made before the filing was begun to indicate to

layer around a core of the second layer, and the layers, a few drawings will make this separation

dissi.

start. The crystal used in this attempt was very Mindly furnished us for the ourgoes by Prof. T. L. Mineralegy, when it was commisted, it did not seem . Luder to and to

were filed off with an ordinary fale. The cryatel .svova hall rozeh sho shr al bodtem aldr ni besu It had been kept cerefully wrouped, and was simply taken and put in a olam and filed. Marks sere

what depth the filing should be carried out in order to remove approximately one third of the crystal.

A rather coarse file was then used and the crystal filed off down to these marks. This material was then used as the outermost layer. The same procedure was repeated and the middle layer thus removed.

The core was then ground up and used.

In using an ordinary file, there is danger of introducing an appreciable amount of iron or steel into the uraninite. In order to take out as much of this as possible, a magnetic separation was resorted to. The ore which had been filed off was placed in a thin layer on a sheet of paper. A strong electromagnet resting on a thin sheet of brass was then connected up. The paper with the ore on it was then brought up very close to the copper sheet and was afterwards lowered. The iron and steel, being magnetic, would cling to the copper while the ore would fall back onto the paper. By repeating this procedure a few times, a considerable portion, if not all of the particles of steel were removed.

The ore was then ground very fine in an agate morter and was then considered ready for use in the analyses.

The core was their ground we and used.

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In mains an appreciable amount of iron or steel into the uraninits. In order to take out as much into the uraninits. In order to take out as much of this as possible, a magnistic aspuration was resorted to. The one which had been filed off was placed in a tain layer on a sheet of paper. A strent electromagnet resting on a tain sheet of paper. A strent then connected up. The reper with sheet of brees was then connected up. The reper with the ore on it was an electromagnet up very ploes to the dopper anest and would fall had offer a considering to the paper. By repeating the ore procedure a few times, a considerable nortion, if procedure a few times, a considerable nortion, if not all of the particles of steel with recention, if

The ore was then pround very fine in an egetaction and me then aconsidered ready for use in
the analyses.

ANALYSIS FOR LEAD, URANIUM, AND THORIUM

In general, the method reported by E. M. Kipp was used in the determination of the lead, uranium, and thorium. However, it was found that in one place at least his method did not give results which were at all satisfactory. He reports that in the separation of lead from uranium and thorium, the precipitation with hydrogen sulfide should be carried out in 4% nitric acid. He says:

"The concentration of the acid should not exceed 4% as uranium will not precipitate as the sulfide in this concentration, and at a higher concentration, some lead may be lost."

According to Kipp's report, the lead is precipitated by passing in a moderate stream of washed hydrogen sulfide for about an hour. (It is interesting to note here that these directions correspond exactly to those given by Dr. J. P. Marble in a private communication except that Dr. Marble reports that the lead is precipitated in 5% acid concentration.)

In running through a known in order to become familiar with the methods, it was found that in exactly 4% nitric acid solution there would be

In senotal, the method reported by E. W. Etpp one uses in the determination of the lead, driving, and therium. However, it was found time in one place at least his method did not give results which were at all satisfactory. He reports that in the separation of lead from oranium and therium, the precipitation with dydrogen suifide should be carried out in 4.5 mitrig acid. He sayor

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except of tebro of needs a decorat animat of the test to be seed that the rest motion and the rest and the settles and the set

practically no lead precipitated. In this case, in order to get the lead to precipitate, a little ammonium hydroxide was added. However, even after the addition of this hydroxide, the results showed that some of the lead had been lost.

In order to determine the lowest possible acid concentration in which the uranium would not precipitate, a little pure uranyl nitrate was dissolved in water and varying amounts of acid added. It was found that in the case of the pure salt dissolved in water, there was no precipitate with hydrogen sulfide. Also there was no precipitate in any case where any acid had been added.

Accordingly, in the next run, the solution upon which the separation was to be carried out was first neutralized with ammonium hydroxide and then two or three cubic centimeters of concentrated nitric acid were added. Washed H₂S was then passed in for a couple of hours. In this case, however, it was found that some of the uranium had been precipitated along with the lead. Thus a slightly more acid solution was needed. The procedure which was used in this run was as follows:

The outside layer which had been carefully

oractionally do lead precisionsed. In this case, in order to get the lead to precipionse, a little square to get the lead to precipionse, even after the addition of this hydroxide, the results about the control to so that some of the lead had been lead.

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The outside layer widch had been carefully

ground up was placed in a clean, dry weighing bottle, and three samples were weighed out by difference. The samples were placed in 250 ml. Erlenmeyer flasks and enough water was added to just cover them.

About 50 ml. of 1:1 nitric acid was added and the flasks heated slightly. The solutions were kept just below the boiling point for four or five hours, or until evolution of helium had stopped. The solutions were then diluted to about 150 ml. and were filtered. The precipitates were thoroughly washed and the residues were ignited in weighed platinum crucibles to constant weight. The results were recorded as per cent silica.

The filtrates contained lead, uranium, and thorium. They were diluted to about 400 ml. and enough ammonium hydroxide was added to make them just neutral. About 2 ml. of redistilled nitric acid was then added. The solutions were then placed in 1000 ml. Erlenmeyer flasks and washed hydrogen sulfide passed in under pressure for three or four hours. A stream of washed hydrogen sulfide was then allowed to flow through the solutions while they were heated to boiling. They were kept at the boiling point for about fifteen minutes and

ground up was placed in a clean, dry weighing bottle, and three samples were weighed out by difference. The exaples were placed in 250 ml. Mrienmayer flasks and enough water was added to just cover them.

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were then allowed to cool slowly.

The precipitated lead sulfide was then filtered off, and the filtrate should then contain the uranium and thorium. The lead sulfide was then washed as completely as possible into an evaporating dish. The flask in which the precipitation had been carried out was washed thoroughly with concentrated nitric acid, and this solution added to the lead sulfide in the evaporating dish. The filter paper which contained the lead sulfide was then ignited, the residue dissolved in nitric acid, and added to the rest of the solution. This whole mixture was digested for several hours with concentrated nitric acid and was then baked almost to dryness.

At this point a decided yellow color was noticed due to the uranium which had been precipitated along with the lead, and these analyses were therefore, disregarded for the time being and some new ones started using the second layer.

The final method which was worked out for the separation of lead and uranium is the one which is given in the following description.

The second layer was then ground up carefully and placed in a clean, dry weighing bottle. Three

were then allowed to dool slowly.

The precipitate should then contain the unnium off, and the filtrate should then contain the unnium and thorium. The lead sulfide was then woshed as completely so possible into an evaporating dist. The flash in which the precipitation and been carried out was weared theroughly with consentrated mitric acid, and this solution added to the lead sulfide in the avaporating dist. The filter paper which contained the lead sulfide was then ignited, the residue dissolved in mitric acid, and added to the rest of the several hours with concentrated mitric said and wet then acked almost to dayness.

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The final method water was verked out for the separation of lead and aremius is the one widen is given in the following description.

The second layer was then ground up carefully and placed in a clean, day weighing bottle. Three

samples were then weighed out by difference and the samples dissolved and the silica determined just as had been done in the first layer. The filtrates which would contain the lead, uranium, and thorium were carefully neutralized and 13 ml. of redistilled nitric acid added. The solutions were then diluted to about 400 ml. which would make the resulting solution about 3% in nitric acid.

The solutions were then placed in 1000 ml.

Erlenmeyer flasks and washed hydrogen sulfide was passed in under pressure for three or four hours.

A stream of washed hydrogen sulfide was then allowed to flow through the solutions while they were heated to boiling and kept near the boiling point for about fifteen minutes. They were then allowed to cool slowly.

The solutions were filtered leaving a residue of lead sulfide and a filtrate containing the uranium and thorium. The residue was thoroughly washed to free it from any uranium or thorium and was then washed as completely as possible into an evaporating dish. The filter paper was ignited, the residue dissolved in nitric acid, and added to this solution. Also the flask in which the precipitation had been

samples were then weighed out by difference and the samples dissolved and the silice determined just as had been done in the first layer. The filtrates which would contain the load, premium, and therium were carefully neutralized and 12 ml. of redistilled nitric acid added. The solutions were then diluted to about 400 ml. which would make the resulting solution about 3" in nitric sold.

The solutions were then placed in 1000 al.

Inlemmeyer flasks and washed hydrogen sulfide was pareed in under pressure for three or four hours. A stream of washed hydrogen sulfide was then allowed to flow through the solutions while they were heated to boiling and kept near the boiling point for about fifteen minutes. They were then allowed to cool slowly.

The solutions were filtered lesving a residue of feed sulfide and a filtrate containing the uranium and thorium. The residue was thoroughly washed to free it from any unanium or continue and was then washed as completely as possible into an evaporating dism. The filter paper was ignited, the residue dissolved in nitric sold, and added to this solution. Also the flack in which the precipitation had been

carried out was rinsed with nitric acid and this was added to the rest in the evaporating dish. The whole mixture was digested for several hours with concentrated nitric acid and was then baked out almost to dryness. It was then dissolved in water and the reduced sulphur filtered off. This residue was thoroughly washed, ignited, dissolved in nitric acid, filtered, and the filtrate added to the main portion of lead nitrate solution.

In the mean time, the filtrate which should contain uranium and thorium and which had now been diluted up to about 600 ml. with the wash water from the first precipitation was again treated with hydrogen sulfide as before. An appreciable amount of precipitate had formed so this was filtered off and subjected to treatment identical to the first precipitate of lead sulfide.

The main filtrate which had now been diluted to about 800 ml. was again treated with hydrogen sulfide, and again a small but appreciable amount of precipitate was formed. This precipitate was filtered off and the same procedure as before repeated. The lead nitrate from this precipitation showed no trace of yellow even when dry so it may

cerried out was rineed with eithic sold and this was added to the rest in the evaporating dish. The whole mixture was ligested for several hours with concentrated mitric sold and was then haded out nimest to dryness. It was then dissolved in water and the reduced salphur filtered off. This residue was thoroughly washed, ignited, dissolved in pitric sold, filtered, and the filtrate added to the main portion of lead nitrate soldtion.

In the mean time, the filtrate which should open tein uranium and thich had now been diluted up to shout 600 ml. with the wash water from the first precipitation were again treated with nydrogen salfide as before. An appreciable amount of precipitate had formed so this was filtered off. and emblooved to treatment identical to the first precipitate of lead sulfide.

The main filtrate which had now been diluted to about 800 ml, was as in treated with dydrogen nulfide, and again a small but appropriable amount of procepitate was formed. This precipitate was formed the name procedure as before repeated off and the name procedure as before repeated. The less nitrate from this precipitation showed no trace of yellow even them dry so it may

be concluded that it would be safe to precipitate
the lead in about 1.5% nitric acid, and that in this
concentration precipitation of the lead would be
complete.

The three lead nitrate solutions were then combined and evaporated to dryness. Two or three ml. of nitric acid were added to keep other salts in solution and the residue was dissolved in 50 ml. water. To this solution was added a slight excess of sulfuric acid. The resulting solution was evaporated to fumes of sulphur trioxide, 30 to 40 ml. water was added, and the mixture allowed to stand overnight. The lead sulphate separates in fine white crystals. The solution and crystals were then transferred to a weighed platinum crucible, the acid fumed off, and the sulphate heated to constant weight over a sand bath.

The lead sulphate was then rinsed out of the crucible with hot 2 normal ammonium acetate. The residue was filtered off and ignited to constant weight. The weight of the lead sulphate is then the difference in the two weights.

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The lead nulphase was then rinsed out of the crucible with hot 2 normal associate. The residue was filtered of and ignited to constant weight. The meight of the lead sulphase is then the difference in the two weights.

SEPARATION OF THORIUM AND URANIUM

The filtrate from the final hydrogen sulfide precipitation was evaporated to about 600 ml. All the excess hydrogen sulfide was driven off by boiling. About fifteen ml. or 1:1 nitric acid were added, and the solution again boiled to remove all traces of carbon dioxide. An excess of freshly distilled ammonium hydroxide was then added, and the precipitated uranium and thorium hydroxides allowed to settle. The solution was kept near boiling and was filtered hot in order to prevent any absorption of carbon dioxide. This precaution is necessary since if any carbon dioxide were absorbed, it would combine with the ammonia to form ammonium carbonate which in turn would dissolve the uranium and thorium hydroxides.

The precipitate was then washed with two per cent ammonium hydroxide after which it was all dissolved in concentrated nitric acid. It was then diluted to about 600 ml., heated nearly to boiling, reprecipitated with freshly distilled ammonium hydroxide, filtered hot, and washed with two per cent ammonium hydroxide. Uranium was recovered from the combined filtrates by evaporating to

absorbed, it would compile with the sampuis to form
sampulum carbonate which in turn rould dissolve the
uranium and thortum hydroxides.
The precipitate was then washed with two per

The precipitate was then where it as all less than one that authorized by the set of the

dryness several times with an excess of aqua regia which would drive off the ammonium salts. The residue was then dissolved in nitric acid, and any uranium precipitated as before.

In both the preliminary run and in the present one the directions given by J. P. Marble and E. M. Kipp were followed at this point. According to both of them, all the uranium and thorium hydroxides were dissolved in nitric acid. This solution was then evaporated to dryness and the residue was dissolved in four per cent nitric acid. This solution should contain about 300 ml. It was then poured into about one-fourth its volume of 10% oxalic acid and allowed to stand for three or four days.

In the preliminary run the above procedure was followed exactly. The precipitates were filtered off, and by the time I was ready to carry out the determination of uranium on the filtrate, there was a very appreciable amount of precipitate on the bottom of the beaker. It was believed that this precipitate was some more thorium oxalate, so it was filtered off and added to the rest of the thorium oxalate precipitate. However, the same thing

dryness several times with an excess of aqua regin which would drive off the immoralism salts. The residue was then dismolved in mivric acid, and any uranium precipitated as before.

In both the preliminary run and in the present one the directions given by J. E. Narble and M. M. Mipp were followed at this point, According to both of them, all the uranium and therium to both of them, all the uranium and therium hydroxides were dissolved in hitric soid. This solution was then evaporated to dryness and the residue was disnolved in four per dent nitric soid. It was then poured into about one-rounth its volume of 10; then poured into about one-rounth its volume of 10; days.

In the preliminary run the above procedure was followed amount. The precipitates were filtered off, and by the time I was ready to carry out the determination of wranting on the filtrate, thate was a very appreciable amount of precipitate on the bottom of the beam, It was believed that this precipitate was some more thorium oxalate, so it was filtered off and added to the rest of the thorium oxalate, as it was precipitate, when added to the rest of the thorium oxalate precipitate. Wowever, the man tidna

happened again so this time the solutions were heated nearly to boiling in a hope that this would help the formation of the precipitate. They were then allowed to stand for a day or two, and were then filtered. All the thorium oxalate seemed to be removed by this procedure. However it must be remembered that by this time the solutions were nearly double in volume since several portions of wash water had been added to them. This would mean that the acid concentration had been lowered a great deal. The last precipitate to be filtered out showed no trace of yellow, so it was assumed that no uranium had precipitated even at this low acid concentration since if it had the precipitate would certainly showed a trace of yellow color.

In the present run great care was taken to make the nitric acid solution in which the uranium and thorium hydroxides were dissolved exactly four per cent since it was feared that an error might have been made in the preliminary run, especially in view of the results. The uranium and thorium hydroxides were dissolved in the carefully prepared nitric acid and this solution was poured into about one-fourth its volume of 10% oxalic acid. The resulting

Auppened agein so this time the solutions were heat the formation of the precipitate. They were then allowed to stand for a day of two, and were then allowed to stand for a day of two, and were then filtered. All the thermalm examines sented to be remembered that by the time time also solutions were remembered that by the time the solutions were nearly double in volume since several portions of that the noid someshies in them and been lower to them. This mould weam that the noid someshies in the deen lowered a great and some of that the noid someshies in add been lowered a great and some and the transpread out should no crace of yellow, so it was assumed that no crace of yellow, so it was assumed that on concentration since if it and the precipitate would containly showed a trace of yellow ould.

In the present run grent care who taken so make the nitric acid solution in which the archium and the nitric acid solution in which the archium and chartes were dissolved exactly four per cent since it was feated that on error might have been made in the preliminary run, superially in view of the results. The archium and thorium bydroxides were dissolved in the archiully prepared mitric sold and this solution was poured into about one-fourth and this solution was poured into about one-fourth its volume of 10 oxalic acid. Whe resulting

solution was about 400 ml. as is required by the directions of both Kipp and Marble. The solutions were then kept near boiling for about one half hour, and were then set aside for nearly a week in hope that this procedure would cause all the thorium to precipitate. The precipitate was then filtered off and treated as described under the determination of thorium.

As the precipitate of thorium oxalate was washed it was noticed that as the wash water went into the beaker containing the filtrate, it formed two layers with the filtrate, and there was a white ring of precipitate formed at the interface. Apparently as the wash water mixed with the filtrate it lowered the acid concentration and more thorium oxalate was formed. This seems to be rather definite proof that if the precipitations were carried out at four per cent acid concentration, not all of the thorium would be precipitated, but some would remain with the uranium and later be precipitated with it. In order to get all the thorium precipitated for this run, the solutions were diluted to about 800 ml. and were set aside for a day. By doing this, the nitric acid concentration was lowered about half, or about to

directions of opin Mips and Marble. The solutions directions of opin Mips and Marble. The solutions were then kept near boiling for enout one half hear, and were then set seide for nearly a meek in hope that this procedure vould cause all the thorium to precipitate. The procedure would cause all the thorium of thested as described ander the determination of therium.

amount containing the filters, it formed two layers with the filtrate, and there was a while ring of precipitate formel at the interface. Apparently as formed. Inte seems to be rather definite proof that dent acid concentration, not all of the thorian would be greetpitered, but some would remain with the uranium and later be precipitated with it, In order , Mul suit tol lededinioarg mulroid and lie se ou the solutions were diluted to about 500 ml. and were set saids for a day. By doing this, the mitric sold

two per cent. The precipitates which formed were filtered off, and were added to the rest of the thorium oxalate. The filtrates were then treated to remove oxalic acid as is described under the determination of uranium. However, after this had been done, it was found that on trying to dissolve the residue in water, there was a very insoluble precipitate left.

This precipitate could not be accounted for at all. The only thing that was at all conceivable was that it was some more thorium oxalate. However, this did not seem possible either since the precipitation had finally been carried out in half the acid concentration required by the directions given by both Marble and Kipp. Therefore, it was decided that one of the precipitates would be sacrificed and a qualitative test run on it. Accordingly, one of the precipitates was filtered off, washed, and the filter paper burned off at a low temperature. The residue was unmistakably the same as the thorium oxalate after such treatment. That is, it was a light buff color, and of about the same consistency, and insoluble in concentrated nitric acid. It was therefore decided that there must still be some

two get oest, the promipitates which formed were filtered off, and were sided to the rest of the filtered of the then treated therium oxalate. The filteredes were then treated to remove oxalic acid so is described under the determination of branium. However, after this had seen done, it was faund that on trying to dispolve the residue in water, there was a very insoluble precipitate left.

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thorium in the solution. All three solutions were therefore neutralized with freshly distilled ammonium hydroxide, and made carefully to one per cent nitric acid concentration. They were then poured into 10% oxalic acid solution as before. This time they were allowed to stand on the steam bath for a day before they were filtered. When they were filtered, the precipitates showed absolutely no trace of yellow which indicated that uranium is not precipitated as the oxalate in a solution which has a nitric acid concentration of about one per cent. It is therefore concluded that it would be best to precipitate the thorium oxalate in about one per cent nitric acid concentration since at this concentration, all the thorium would be precipitated, but no uranium. After the complete separation had finally been made, the procedure was followed as described below.

the same in the selection. If A . noise of the continue area tor one of yillifers o blem hate, shinoungi muito out cont sivile sold concentration. They were tron poured into low oralle and solution as before, bath for a day before they were filtered, when auch one per cent, it is therefore concluded that be precipitated, out no uranium. After the complete seneration had finally been made, the procedure was followed as described below.

DETERMINATION OF URANIUM

The thorium and other rare earth oxalates were filtered and set aside for later analysis: the filtrates contained the uranium. The filtrates from the oxalic acid treatment were evaporated almost to dryness on a hot plate. It was found best not to evaporate to the formation of crystals because if this were done very bad spattering would occur. Therefore, the filtrates were evaporated only to small volume. An excess of concentrated nitric acid was then added and the solutions evaporated to small volume several times. This treatment was continued until no more red-brown fumes were given off. This indicated that all the oxalic acid had been decomposed. The final uranium solutions should be a clear, light golden yellow in color, since sometimes the red fumes will stop coming off before quite all the oxalic acid is gone and this will leave the solutions colored slightly redish.

When all the oxalic acid was gone as shown by the absence of red-brown fumes on heating with concentrated nitric acid, the solutions were diluted with water to several hundred ml. (about 400 ml. in this case). Ammonium hydroxide in which was

DESCRIPTION OF PERSONS ASSESSED.

filtrates contained the dramium. The filtrates almost to dryrues on a het plate. It has found occur. Therefore, the filtrates were evaporated only to small volume. An excess of concentration oratel to small volume several times. This treatwere given off. This indicated that all the orallic sold had been decomposed. The first arent on solutions should be a clear, light golden yellow to color, will leave the solutions colored slightly redish,

the absence of red-brown funcs on heating with concentrated mitric soid, the solubions were diluted with water to several number of all (about 100 ml. in this case). Ammonium hydroxids in which was

dissolved a considerable quantity of ammonium carbonate was then added to a slight excess beyond the methyl red end point. A small lump of solid ammonium carbonate was then added. The solutions were then warmed gently until carbon dioxide began to be evolved. The solutions were removed from the flame, and any precipitate was allowed to settle. The precipitates, consisting chiefly of iron and aluminum, were then filtered off and were washed thoroughly with a dilute, about one fourth saturated, solution of ammonium carbonate. Finally, the precipitates were washed with hot dilute ammonium chloride until they did not smell of ammonium carbonate. The precipitates were then dissolved and reprecipitated as before. The combined filtrates were then evaporated to dryness several times with excess aqua regia to remove the ammonium salts. Care was taken to avoid loss by spattering when the acid was added. The residues were then dissolved in water, and diluted to about 350 ml. Another precipitation with ammonium hydroxide and ammonium carbonate was then carried out. The precipitates were filtered off, and were thoroughly washed as before, but were not reprecipitated. The filtrates

the metryl red and point. A small lump of salid amonium carbonate was then adied. The mointions to be evolved, the columnions were removed from the flame, and any precipitate was allowed to settle, aluminute, were tren 'll tered off and were washed solution of manorium exchonate. Finally, the derionate. The precipitates were then Albertved and reprecipitated as before. The commined filtraics excess agus regis to remove the sumonium asile. acid was added. The residues were then dissolved TondonA . in Occ thods of hetulis bus , redsw pr estationate was then carried out. The precipitates before, but were not reprodupitated. The filtrates

were then freed of ammonium salts as before. This process removed practically all the iron and aluminum.

It is interesting to note here that there was a considerable quantity of iron present in spite of the fact that when the uranium had been precipitated as hydroxide previously it appeared a pure canary yellow in color. Always before it has been believed that if the uranium came down a pure canary yellow as a hydroxide, no iron was present.

After the removal of ammonium salts, the solutions were diluted to about 500 ml. and the carbon dioxide driven off by boiling. The uranium was then precipitated with a slight excess of freshly distilled ammonium hydroxide and filtered. The precipitate was then dissolved in nitric acid, diluted, and reprecipitated. Traces were removed from the combined filtrates as before. The entire precipitate was then charred and ignited to constant weight in a platinum crucible. This is best done by placing the crucible in a slanting position with the cover tipped across the mouth. The paper is then charred, and with the cover removed, is smoked off. The crucible is then placed in an upright

process removed practically all the tree ont

It is interesting to note here that where was a considerable quantity of iron prosent in apite of are fact that when the uranium had been precipitated as hydroxide previously it appears a pure canary yellow in color. Always before it has been believed that if the uranium come down a pure canary yellow as a hydroxide, no iron was present.

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position and is roasted in the full flame of a meker burner. The material in the crucible should be $\rm U_3O_8$ and there should be no trace of yellow present.

DETERMINATION OF THORIUM

The filter paper from the precipitated thorium and other rare earth oxalates was burned off at a low temperature. The residue was then brought to constant weight with a meker burner. The mixed oxides were then fumed to dryness with 10 ml. portions of concentrated sulfuric acid until they were pure white in color. They were then dissolved in concentrated nitric acid and diluted to 100 ml. This solution was then neutralized with freshly distilled ammonium hydroxide. The thorium was then separated from the other rare earths by precipitation as thorium sebacate with sebacic acid. thorium solutions were heated almost to boiling as was the almost saturated solution of sebacic acid. The sebacic acid was then added in slight excess to the thorium solutions. Any precipitated sebacic acid was washed from the thorium sebacate by means of hot water. The thorium sebacate was then dissolved in nitric acid and reprecipitated until pure white in color. The thorium sebacate was then charred and ignited to constant weight as ThOo with a meker burner.

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low ismersture. The residue was then broncht to constant velicht with a meker barner. The mixed -rac . In Ol Will agentum of hear's gent stew ashing pure white in color. They were then disadved in . In OOL or beinlih has bins sittin hereigneones distilled amount on the character and character than tion as thorner bardes with sensoic act. The . Line viocise to nothilve before to Jeomis and sav olusies heruricione, hay precipitation marteri ent of of hot water. The thorium sabsdate was then ALSwrite in color. The thortum secaps of estima . Tentud Teslem a niti

EXPERIMENTAL RESULTS

Possibly the most interesting and most important results are in the nature of corrections to be made in the accepted procedure for the analysis rather than in the nature of a lead ratio. It was found that both the important separations had to be carried out in a lower acid concentration than was recorded by E. M. Kipp. Thus in the precipitation of lead as lead sulfide, it was necessary to carry out the precipitation in about 1.5 per cent nitric acid concentration rather than in about 4 per cent as was previously reported. In fact in exactly 4 per cent nitric acid concentration, it was found that the lead would not even precipitate appreciably. In the case of the separation of thorium and the rare earth oxalates from the uranium, it was found that if the precipitation was carried out in about 4 per cent nitric acid, the thorium was very definitely not all precipitated. In this separation, the best conditions seemed to be with about 1 per cent nitric acid concentration when all the thorium and rare earth oxalates would precipitate, but no uranium oxalate.

As a result of the analyses, the following

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rather than in the nature of a lead rolls, it was recorded by 6. M. Pipp. Thus in the precipited on of lead of these salinds, it was necessary to carry out the presiptables in about 1.0 per sens nittle as as previously reported. In fact in exactly deper cent nitric acid concentrations, it was rough . Alvelosings stavigious nevs con figor heel and send nitely not all predictors. In this separation, urshium oxclate.

As a result of the anolyses, the following

per cents of silica and lead can be reported for this particular crystal of Wilberforce uraninite:

Outside Layer

Silica Determination

1 2

Weight sample .85595 gm .87980 gm

Weight silica .01664 gm .01875 gm

% silica 1.94% 2.13%

Average % silica 2.04%

Middle Layer

Silica Determination

	1	2	3
Weight sample	.69845	.67089	.72261
Weight silica	.00450	.00635	.00677
% silica	0.64%	0.95%	0.94%
Average % silica	0.84%		

Lead Determination

	1	2	3
Weight sample	.69845	.67089	.72261
Weight PbSO ₄	.10557	.09795	.10659
% lead	10.33%	9.97%	10.08%
Average % lead	10.13%		

Due to the difficulties encountered in experimental procedure, the work is not yet complete.

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Silice Determination

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Average N lead 10,15%

Due to the difficulties encountered in experi-

mental procedure, the work is not yet doupleve.

Before it is complete, the amount of silica, lead, uranium, and thorium in the outside layer, the middle layer, and the core will be determined by the method given previously. From this data, the lead ratios will be calculated, and it is believed that this will bring forth very good evidence as to whether or not leaching does occur, and if it does, how the most reliable lead ratios can be determined.

defore it is complete, the smoont of ellice, list, ursativa, and conting in the outpite layer, the middle layer, and the core mill be determined by the method given previously. From this date, the lead ration will be celestated, and it is believed that this will bring forth very good evidence as to whether or not leadming does occur, and if it does, how the most reliable lead ration out to determined.

SUMMARY

- 1. The four chief methods of determining the age of the earth have been pointed out and described briefly. The four methods are (1) method based on thickness of strata, (2) method based on age of the ocean, (3) method based on astronomical data, and (4) method based on radioactive disintegration.
- 2. The method which depends on the lead-uranium ratio of radioactive minerals has been discussed to some extent. The various requirements of the method in order to get a reliable ratio have been pointed out. Also, it has been shown how leaching is a possible source of great error in this method.
- 3. The deposits of uraninite at Wilberforce, Ontario, have been described, and the results obtained by other workers using this mineral have been given.
- 4. Changes in the experimental procedure have been found necessary at the two important separations in the analysis. In the case of the precipitation of lead as the sulfide and the precipitation of thorium as the oxalate, it was found that a lower nitric acid concentration than that previously reported was necessary in order to get complete

1. The four chief methods of determining the age of the earth wave oven pulmed out and lescribed bristly. The four deviade are (1) method beand on this trickness of strete, (2) method based on age of the ocean, (3) method used on astronmical data, and (4), method cased on radioactive disintegration.

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4. Changes in the experimental procedure have been found necessary at the two important neparations in the analysis. In the case of the precipitation of lead as the salfido and the precipitation of the oxilate, it was found that a lower nitric and someomeration than that previously reported was necessary in order to get complete.

separation. Also at these lower acid concentrations it was found that in neither case was any uranium precipitated.

- 5. Another very interesting fact was found in that whereas it had always been believed that if the uranium hydroxide came down a pure canary yellow, it contained no iron, actually it was found that in the case of this analysis, although the uranium hydroxide did precipitate a pure canary yellow in color, it did contain a very appreciable quantity of iron.
- 6. The differences in the percentages of silica in the outside and middle layers of the crystal would indicate that the outside layer had undergone much more alteration than the middle layer.
- 7. An outline of future work with this crystal which has been mechanically separated into three layers has been given.

separation. Also at these lower and nonnestions to was found that in neither oase was any araniam precipitates.

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VI Report of the Comittee on the demandant of tol Report of the State of the State

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The author also wants to thank Prof. L. C. Graton, Prof. Charles Palache, and Mr. Harry Berman for their work in cutting the crystal with the diamond saw.

ACCOUNT ADMINANTS

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